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FINAL PLUTONIUM VOLITIZATION REPORT FOR THE PLASMA FURNACE VITRIFICATION TREATABILITY STUDY

Rocky Flats Plant

U. S. Department of Energy
Rocky Flats Field Office
Golden, Colorado

Environmental Restoration Program

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1.0 TEST SUMMARY

In order to ensure that the technical approach proposed by Lockheed for the Pit 9 remediation is viable, key elements of the technology were demonstrated in a series of proof-of-process (POP) tests. These tests included plutonium solubility in triethylamine, nitric acid leach of soil contaminant surrogates and recovery in the counter-current ion exchange (CCIX)/chemical extraction system, PCF-6 (plasma centrifugal furnace) 100-hour melter operations test, bench-scale melter plutonium volatilization test, maintenance-in-containment test of the plasma melter, and testing of the dig face monitor. This report presents the results of the Pu volatilization POP test.

During Pit 9 remediation, Lockheed will use a plasma melter to treat wastes and soils contaminated with organics, transuranic nuclides, and heavy metals. The waste streams are fed into a sealed centrifuge, where they are exposed to intense heat from a transferred-arc plasma torch. Organic material will be destroyed almost immediately upon entering the primary chamber. Off-gases pass through a secondary combustion chamber and are cleaned in an off-gas treatment system.

Inorganic material is reduced to a molten phase that is uniformly heated, mixed, and exposed to the oxidizing environment of the primary chamber by the motion of the centrifuge and turbulence from the plasma arc. When the centrifuge is slowed, the molten slag is discharged through the throat in the bottom and collects in a mold or drum in the slag collection chamber below.

Testing has demonstrated that the slag meets all Toxicity Characteristic Leaching Procedure (TCLP) standards for leachability and that the process has a destruction removal efficiency (DRE) greater than 99.999 percent for soils containing metals and organics. Theoretical calculations indicate that all or nearly all plutonium will remain in the slag during processing of soils contaminated with radioactive materials, but these calculations have not been tested by experimentation. The purpose of the POP test, which will be performed in a bench-scale furnace, is to determine the percentage of plutonium that will volatilize and to correlate the data from this POP test to the 100-hour operations test.

1.1 Plutonium Volatilization Test System

The complete bench-scale furnace system is shown in Figure 1-1. Sub-units of the furnace test system are described below.

1.1.1 Furnace Description

The bench-scale furnace system is a 150-kilowatt (kW) laboratory furnace similar to those manufactured by Retech for material alloy studies. This furnace is already constructed at Retech, Inc. and will be modified for the volatilization test. The furnace system includes a melt chamber, plasma torches, plasma torch manipulator, power supplies, hydraulic power unit, water manifold, closed water cooling system, and manual controls mounted on a skid.

Picture not available

Figure 1-1 Bench Scale PCF Schematic

In addition to the basic furnace, Retech is providing a flexible glovebox enclosure, rotating hearth, material feeder, and closed-loop gas system.

Melt Chamber

The melt chamber is approximately 31 inches in diameter and 30 inches long and is mounted horizontally on legs. It is constructed of mild steel, is water cooled, and contains two view ports, one on the chamber side and one on the door. The interior wall of the chamber is polished to enhance the recovery of any material deposited on the walls.

Chamber temperature near the wall during operation will be lower than in a refractory-lined plasma centrifugal furnace. The temperature of the slag will rise to 1,200 to 1500 C°, which is comparable to that of a full-scale PCF. Gas temperature over the hearth is 800 to 1,000 C. The bench-scale furnace will operate at slightly negative pressure of 9 to 15 inches of water gauge (vacuum), which is also comparable to PCF-6 and PCF-8 operation. A view into the melt chamber after test 3 is shown in Figure 1-2.

RP75T Plasma Torch Assembly

The RP75T plasma torch utilizes electric energy to ionize gas introduced through the torch to produce a conductive plasma arc. To start the torch, a low-energy plasma arc is initiated between the electrode and the exit nozzle of the torch.

The torch is mounted at the end of a water-cooled torch ram. All gas, torch cooling water, and power hoses pass up through the ram and out the top of the ram. The torch ram passes through a sealed ball joint mounted in a flanged, water-cooled housing. The bearing set for the torch swivel ball joint is non-conductive, making the torch ram electrically isolated.

Plasma Torch Manipulator

The manipulator assembly is mounted on top of the melt chamber. It holds the torch ram and moves the ram within the chamber. The manipulator assembly consists of an *x-y* plate and one hydraulic cylinder each for the *x* and *y* axes and two cylinders for the *z* axis (four total). Vertical (*z*) travel is approximately 12 to 16 inches. Horizontal (*x-y*) motion allows the torch to rotate about the vertical center line up to 15° from vertical. Each hydraulic cylinder is controlled by a four-way valve. A joystick and rocker switch on the control panel allow the operator to change the termination point of the arc in the rotary hearth.

Glovebox

To allow access to the chamber interior without loss of containment, a flexible glovebox enclosure will be attached to the chamber after it has cooled below the temperature allowed for the flexible enclosure material. This allows sample and hearth removal as well

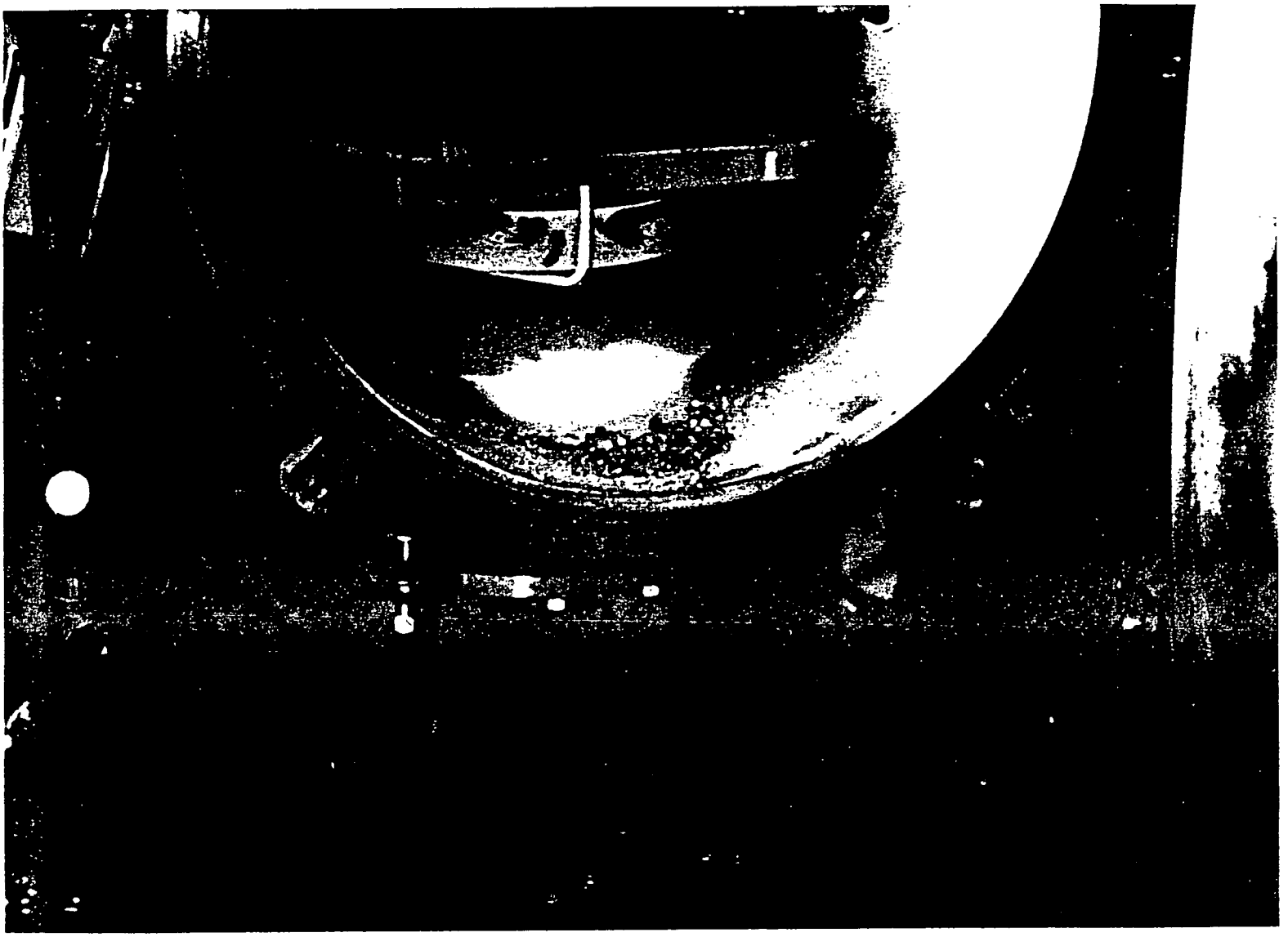

Figure 1-2 Melt Chamber Open After Test 3

as torch maintenance if necessary. A bag will be used for loading the material feeder. The melt chamber glovebox is shown in Figure 1-3.

Hearth

The rotary hearth is designed to be as simple as possible and light enough for the operator to remove from the melt chamber. The rotary hearth will be constructed from graphite and refractory. Graphite was selected because it is the best conductive, high-temperature material available for construction of the hearth. Tests conducted at Retech have demonstrated a very short life for steel and refractory hearths. It is anticipated that a new hearth will be used for each melt test. Tests conducted at Retech have demonstrated that approximately 90 grams (g) of carbon will combust from the hearth during each test run. The oxygen content proportional control within the closed-loop gas system has been designed to account for this.

The rotary hearth rests on a water-cooled hearth plate bolted to the rotation mechanism. Rotation of the hearth distributes the molten slag around the hearth and allows a continually varying arc termination point on the slag, as allowed on a full-size PCF. The distribution of slag facilitates oxidation of metals and uniform heating and mixing of the slag. The rotation mechanism is driven by a frequency-controlled AC electric motor activated from the control panel. Operating speed is expected to be 20 to 40 revolutions per minute (rpm). Removal of the hearth after test 3 is shown in Figure 1-4.

Material Feeder

The material feeder holds material and allows the operator to feed material into the hearth. The feeder consists of a chamber containing a magnetic vibrator, tray, and chute. To load the feeder, material is "bagged" into the feeder port. The material is placed on the feeder tray, and the operator closes the door. The door is then wiped, and the bag is removed. To begin feeding the material, the operator cranks the chute over the hearth and turns on the vibrator to feed the material. A stop ensures consistent positioning of the chute. The feeder with glovebox attached on top is shown in Figure 1-5. Operation of the feeder during test 3 is shown in Figure 1-6.

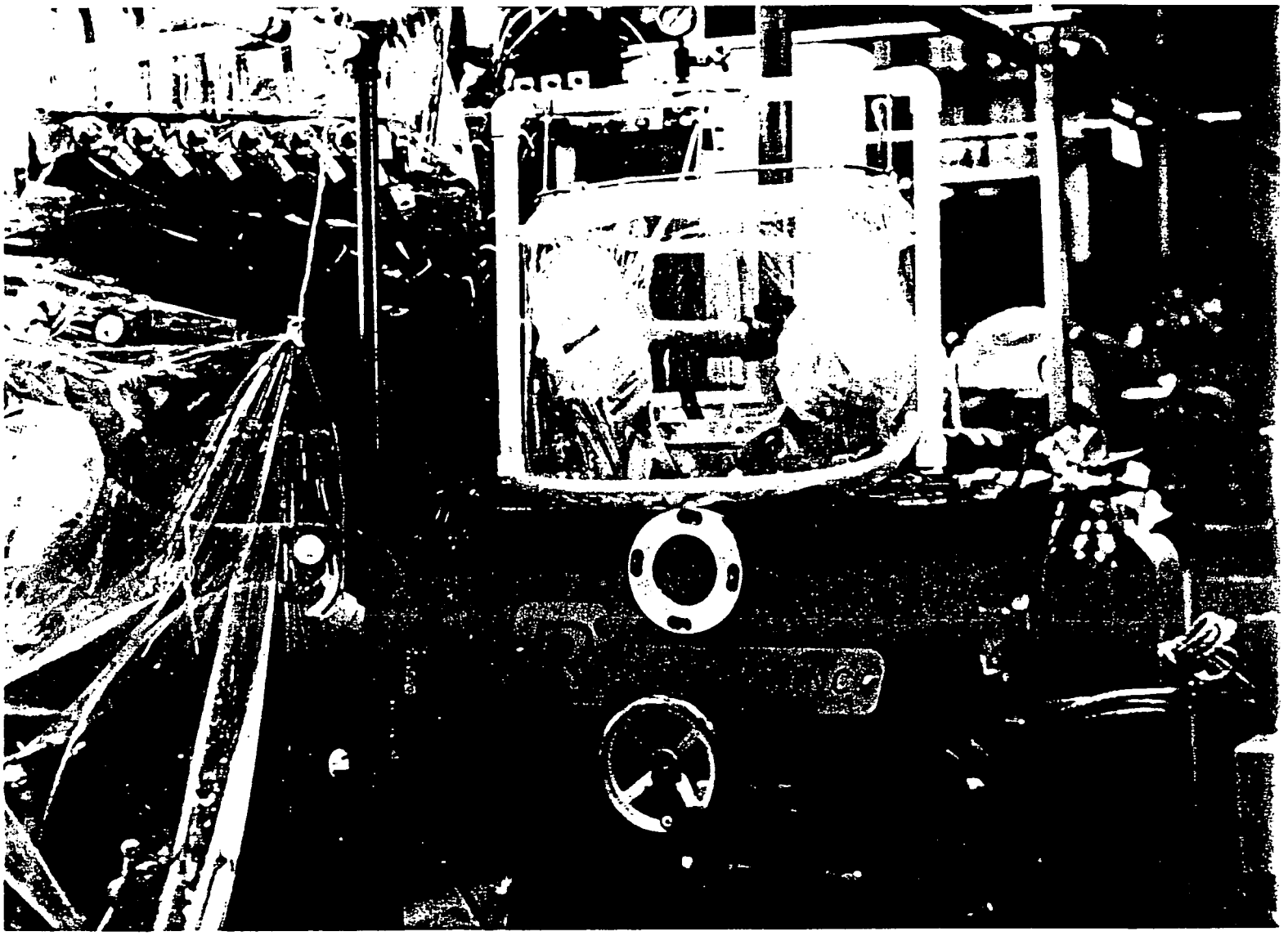

Figure 1-3 Test Apparatus at Retech. Ready for Tests 1 & 2

Figure 1-4 Post-Melt Hearth Removal, Test 3, TREAT

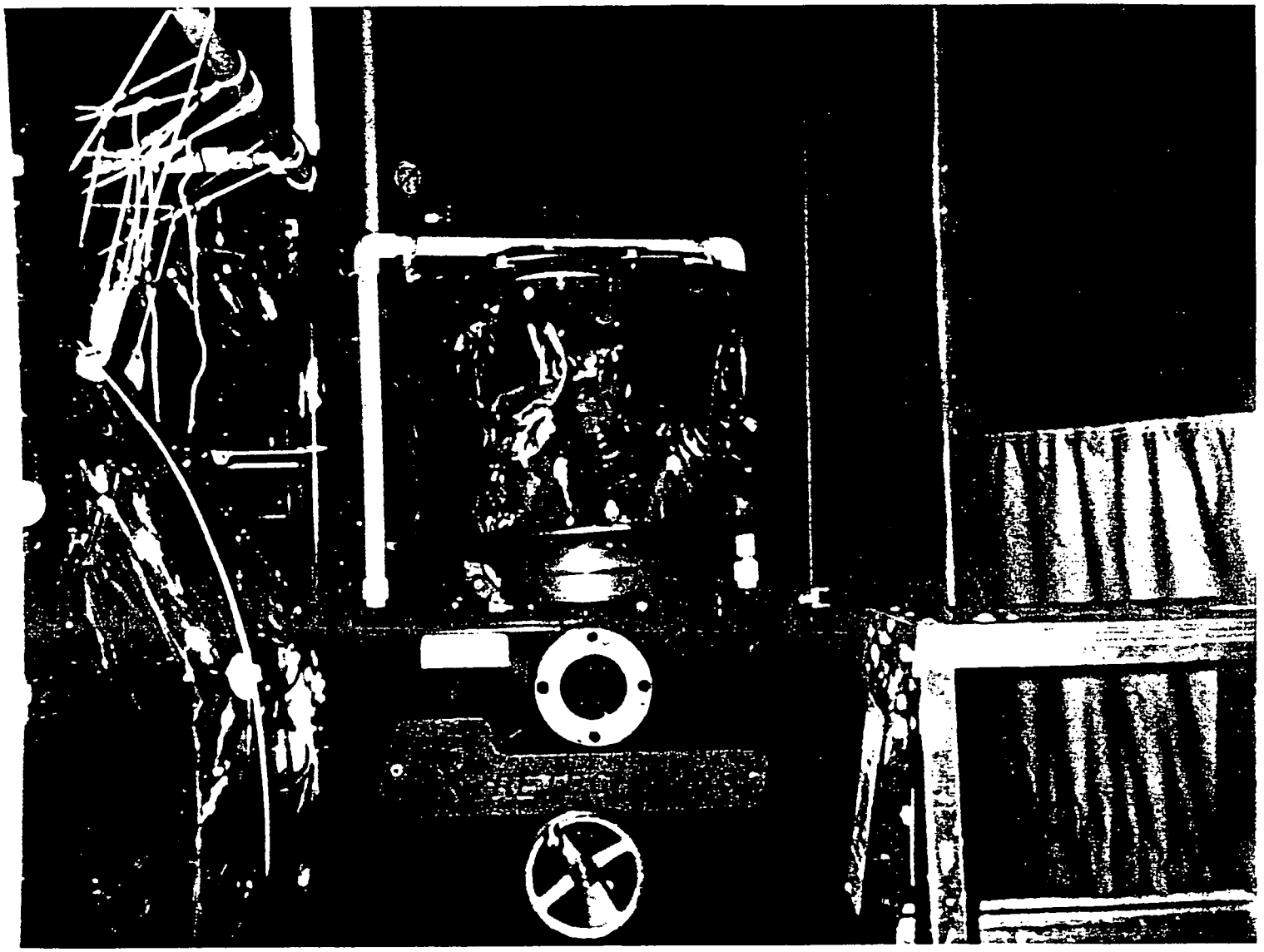

Figure 1-5 Glove Box for Soil Feeder, Tests 3 & 4

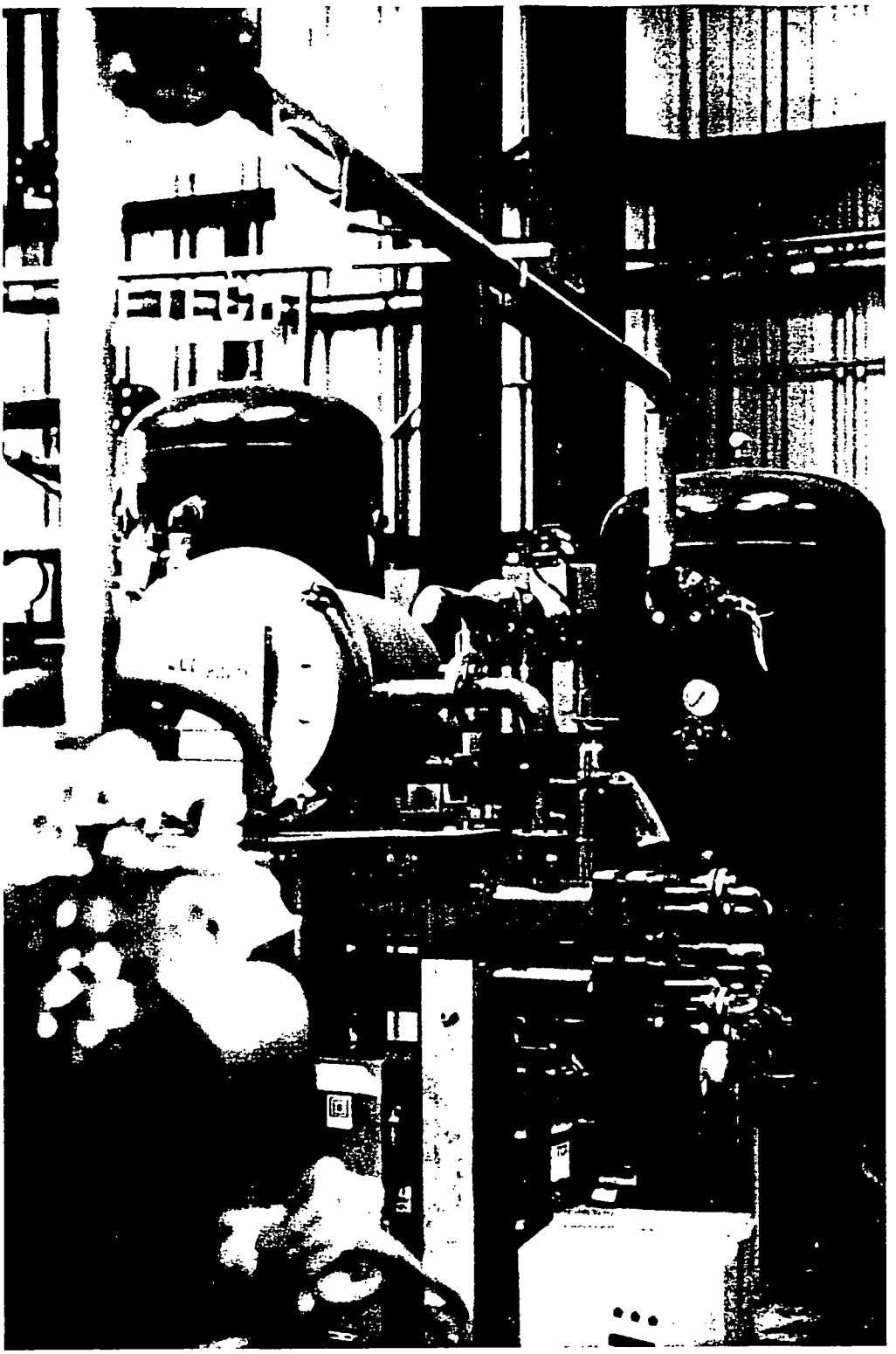

Figure 1-6 Feed Soil Loading, Test 3

Closed-Loop Torch Gas System

The closed-loop gas treatment system cleans, cools, and recirculates furnace gas through the torch, view ports, and gas port in the chamber. The purpose of the closed-loop system is to eliminate emissions. Storage bottles will be supplied to receive excess gas from operation in a sealed, safe manner.

The system is modeled on closed-loop gas systems for metallurgical furnaces and was tailored to meet the requirements of the volatilization tests. System gas is predominantly nitrogen but will include oxygen controlled to 12 ± 3 percent using a proportional control and carbon dioxide from combustion of small amounts of organic material in the system. Gases will also include helium from torch starting and trace amounts of carbon monoxide and NO_x. The system is described as follows:

> The primary gas flow from the bench-scale furnace is drawn by a water-cooled, inter-cooled two-stage gas transfer compressor through a cold finger, the oxygen analyzer probe housing, and then through the high-efficiency particulate air (HEPA) filter. From the HEPA filter, the gas flows through a thermal mass flow meter through the carbon adsorption unit and then through the compressor to a receiver. Vacuum in the primary chamber is controlled by throttling the inlet of the compressor using a proportional control to meet the 12"±3" vacuum in the chamber, as measured by a Setra pressure transducer. The receiver is sized to allow the torch to run from bottled torch gas or on recirculated gas. In either mode, gas flow through the chamber is controlled by manually setting needle valves at the chamber and verifying flow using the rotameters. When set, the gas flow through these rotameters is expected to be relatively constant throughout the test.
>
> The closed-loop system also includes an emergency bottle that is sized to capture the furnace volume at a relief valve pressure of 2 psig nominal. Prior to each run, the emergency bottle is evacuated using the vacuum pump supplied for this purpose. The vacuum pump will also be used to leak test the system up to the HEPA filter before plutonium melt tests are performed. The closed loop system is shown in Figure 1-7.

Plasma Power Supplies

Two 75-kW MacroAmp direct current, saturable reactor power supplies are used for the volatilization test and are designed specifically for the RP75T plasma torch. They provide the electrical current for the main plasma arc from a high-voltage or lower voltage tap, depending on the type of gas run through the torch. The arc voltage is expected to be 300 to 400 volts DC.

Picture not Available

Figure 1-7 Closed Loop Gas System, Test 3, TREAT

Hydraulic Power Unit

A small hydraulic unit is located beneath the melt chamber. This unit supplies hydraulic pressure to the torch manipulator cylinders.

Cooling Water System

The bench-scale furnace is equipped with two cooling water systems. The closed water system provides cooling water to the plasma torch. This closed system consists of one pump and a tank that provides 3 gallons of water per minute of demineralized or distilled water at 150 pounds per square inch (psi) (minimum) to the torch electrode and body. Sufficient water flow prevents heat damage to the internal parts of the torch and is essential for operation. Other water circuits are designed to operate on plant water, but they will be plumbed to a tank to recirculate water in the Lockheed Remediation Testing Laboratory (RTL) to preclude unregulated water release.

Controls

A control console located near the melt chamber allows the operator to observe both the interior of the melt chamber and the control instrumentation during operation. Control switches and meters include the following:

- Power supply
- Microcurrent meter
- Arc voltage meter
- Torch gas pressure switch
- Power supply on/off switch
- Vertical *z* rocker switch
- Torch start switch
- Closed water pump on/off switch
- Torch *x-y* joystick
- Hearth rotation on/off switch
- Hydraulic pump on/off switch
- Gas recirculation control
- Water interlock lights
- Hearth rotation motor controller

A small programmable logic controller interlocks the controls to ensure safe operation of the torch and power supplies. Fluke Hydra 2620A Data Acquisition System (DAS) collects data. The DAS is shown in Figure 1-8.

Figure 1-8 Bench-Scale Furnace, Data Acquisition System

Containment Structure

For Tests 3 and 4, which were conducted with plutonium contaminated soil, the entire furnace and off-gas containment system were installed in a sealed containment structure. The structure, made of Hypalon and measuring approximately 10 feet wide by 12 feet high by 30 feet long, was intended to provide containment of radionuclides in the event of release. No release of contamination from the test apparatus occurred during Tests 3 and 4. The containment structure is shown in Figure 1-9. Contamination control procedures for test 3 are pictured in Figure 1-10.

1.2 Test Description

Four bench-scale plasma melter tests were run during the plutonium volatilization POP test. The POP test consisted of the following four bench-scale melts:

Test 1. Surrogate (cerium oxide) spiked feed soil, melter operated under normal (oxidizing) conditions

Test 2. Iron and polyvinyl chloride (PVC) added to surrogate spiked soil, melter operated under upset (reducing) conditions

Test 3. Plutonium-contaminated feed soil (approximately 1 to 2 nanocuries per gram [nCi/g]), melter operated under normal conditions

Test 4. Iron and PVC added to plutonium-contaminated feed soil, melter operated under upset conditions

1.3 Test Objectives

The plutonium volatilization POP test had two objectives. First, the test measured the percentage of plutonium that volatilized from melter feed soils in a representative plasma processing environment. Second, the test generated operational data for correlation with the 100-hour operations test and engineering data for off-gas system design.

1.4 Test Set-Up and Procedural Summary

For the volatilization test, four tests were run in the plasma bench furnace under conditions that will approximate conditions in a full-scale PCF. The first two tests were performed using transuranic (TRU) surrogates. The last two tests were performed using plutonium-contaminated soil.

After each test, the slag, slag spatter, HEPA filter, and wipes of all surfaces prior to the HEPA were analyzed to determine the percentage of surrogate or plutonium volatilized. The first two tests were conducted at the Retech facility, and the last two tests were conducted at Argonne National Laboratory-West.

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Figure 1-9 Containment Structure at TREAT, Tests 3 & 4

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Figure 1-0 Contamination Control, Test 3

1.5 Test Results Relative to Objectives and Acceptance Criteria

Test results demonstrated that (1) plutonium containment can be achieved in a plasma melting environment, (2) less than 1 percent of plutonium fed to the furnace was volatilized, (3) 100 percent of the plutonium mass can be accounted for (thus exceeding the POP criterion of 90 percent mass balance), and (4) cerium is a suitable surrogate for predicting plutonium behavior in a plasma melting environment. The tests also generated engineering data applicable to design of the off-gas system for the full-scale production melter. All objectives and acceptance criteria of the plutonium volatilization test were successfully met.

2.0 TEST DESCRIPTION

Four tests were conducted to determine the extent of metals volatilization that can be expected during remediation of Pit 9 soil. The first two tests were performed using the same TRU surrogate (cerium oxide) that will be used during the 100-hour operations test. This provided a good correlation of results between this plutonium volatility test and the 100-hour test, as discussed below in Section 4.2.3.

The third and fourth tests were performed with approximately 1.8 nCi/g plutonium-contaminated soil. The first and third tests were conducted under typical operating conditions in the melter (an oxidizing environment with a low metals content). The second and fourth test were performed under "envelope" conditions, using soil containing a high content of metals and organic chlorides. The soil recipes are provided in Appendix I of the Comprehensive Test Plan.

To perform each test, approximately 2 kilograms (kg) of uncontaminated soil were placed in the centrifugal hearth of the melter. The torch was started, and the soil was heated until molten. When the soil became molten, approximately 2 kg of soil contaminated with surrogate or plutonium were added to the molten slag using a continuous vibratory feed system. After feeding was completed, the torch continued to dwell on the soil until it was fully mixed.

The closed-loop gas system was in operation throughout the melt test to eliminate gas emissions. It captured some of the volatilized metals in a water-cooled baffle tube (called a cold finger). Volatilized metals that did not condense on the furnace wall or cold finger were trapped by HEPA filtration. Excess gas or water produced during sample processing was collected, analyzed, and treated or released after verifying that any contamination present was below levels allowed for unrestricted release.

A flexible enclosure was fitted over the furnace door for the plutonium tests to eliminate the possibility of spreading contamination. The rotating hearth was removed, sealed in a bag, and then removed for analysis of the slag in the Lockheed Analytical Laboratory (LAL). The cold finger was also removed from its housing to a containment area fitted with a flexible enclosure and cleaned with wipes. The wipes were also sent to LAL for analysis. The HEPA filter was bagged out of its housing and tested. A new HEPA was provided for each test. The remaining system components were sampled or examined for contamination before the next test was conducted.

2.1 Objectives of the Plutonium Volatilization Test

The objective of the plutonium volatilization test was two-fold. First, it determined the percentage of plutonium that will volatilize from the feed soil and the percentage that will remain in the slag in a representative plasma processing environment. (If the volatility of plutonium been significantly greater than theoretical calculations, a plan would have been provided to show how plutonium would be captured in the off-gas system and stabilized.)

Second, the test generated operational and volatilization data to allow correlation with the 100-hour operations test.

2.2 Plutonium Volatilization Test Acceptance Criteria

The test was considered successful because it met the following objectives:

- Sufficient data were generated to quantify the volatility of plutonium during plasma melter operations.
- The tests accounted for the behavior of the surrogate to allow appropriate correlation to the 100-hour operations test (see discussion of this correlation in Section 4.2.3).
- At least 90 percent of the plutonium was accounted for in performing the mass balance.

2.3 Test Experimental Design Basis

This test was designed to determine whether plutonium will volatilize when plutonium-contaminated soil is plasma melted in the PCF-8 intended for remediation of Pit 9. The centerline temperature of the arc, although unmeasurable, is much greater than the temperature required to volatilize, decompose, and ionize essentially all compounds of concern.

Ordinarily, only the torch gas reaches arc temperatures and compounds such as the oxides of silicon, iron, aluminum, and chromium remain almost completely in the slag. The slag reaches a bulk temperature of 1,200 to 1,650°C, depending on density and composition. A small fraction, usually 1 or 2 percent of the total weight of material fed into the melter, is carried through the throat entrained in the gas as very fine particles and is removed by the off-gas treatment system.

Because the vapor pressure of aluminum oxide at 1,650°C is approximately 10^{-6} atmosphere and the vapor pressure of plutonium oxide is approximately 10^{-7} atmosphere[3], little volatilization is expected, provided that the formation of chlorides is prevented. A more complete discussion of the vaporization temperatures and volatility of compounds important to this test is presented in *Plutonium Processing in a Plasma Processing Environment*, Clarence Whitworth, December 20,1991.

The selection criteria weighed the need for data against the cost of performing the tests, the cost of analysis, and the cost of containment and decontamination. Because the calculations indicate that plutonium behaves in a manner similar to known metals, as explained above, the tests provided confirmation of the calculations rather than new data on an unknown. Therefore, four tests of representative soil mixes were selected. PVC (C_2H_3Cl) was included in Tests 2 and 4 for the purpose of providing a halogenated

hydrocarbon. It was added to the feed to provide approximately 1 percent of feed mass as chlorine, which matches the characterization of Pit 9 wastes and the composition of the simulated Pit 9 feed for the 100-hour test. This provided a good correlation of results between this test and the 100-hour test.

The third and fourth tests were performed using plutonium-contaminated soil (approximately 1.8 nCi/g). The first and third tests simulated typical conditions in the melter, namely an oxidizing environment with a low metals content. The second and fourth tests simulated "envelope" conditions, using soil containing a high content of metals and chlorides.

The fate of the surrogate in the 100-hour test was compared to surrogate location in the plutonium volatility test for each set of feed chemistry conditions The comparison of metal location expressed as the percentage of initial feed mass allowed prediction of the fate of plutonium in a full-scale PCF.

The decision to use the bench-scale furnace for this test was based on the furnace's ability to provide a small, safe containment of the PCF process. The bench-scale furnace system will be decontaminated and used for additional slag chemistry and process studies. Although the bench-scale furnace is a cold-wall system, as opposed to a refractory-insulated furnace like the PCF, the energy flowing through the furnace system follows comparable paths. The bench-scale furnace is over-powered on both a volume and surface area basis, when compared with the full-scale PCF equipment.

Correlation of Physical Sizes and Mass Flows

The most important variables with regard to the volatility of plutonium are consistent throughout the three plasma systems being considered (i.e., the bench-scale, the PCF-6 used in the 100-hour test, and the PCF-8 proposed for full-scale production). Chamber pressure and oxygen content are held to the same values (12" water gage nominal and 12 percent oxygen by volume). Plutonium-contaminated or surrogate-spiked soil with or without organics is fed onto a molten slag bed to be rapidly heated and mixed by the plasma torch gas at the arc termination point. Because the bench-scale plutonium volatility furnace is cold walled, the chamber bulk gas temperature is lower than that in a refractory-lined PCF. Bulk gas temperature in the bench-scale furnace is expected to reach the lower end of PCF gas temperatures before the end of each test. Because volatility depends primarily on the temperature of the arc and the slag at the arc termination point, lower bulk gas temperatures within the bench-scale furnace should have only a minor affect on plutonium volatility.

The bench-scale furnace was constructed by Retech using an existing design. The chamber volume and torch installation were predetermined by this design. The ability to feed soil, the rotary hearth, and the oxygen and chamber pressure controls were adapted from existing designs specifically for the plutonium volatility test. The hearth size was selected to allow feeding of a small quantity of material onto a small slag bed, to allow minimum processing time, and to produce minimum test resultant waste. Slag mass per

unit slag surface area represents a compromise to allow processing the small quantity of soil in the bench-scale furnace and is approximately comparable to the PCF-6 and PCF-8.

The features of the PCF-6 represent expected conditions during the simulated Pit 9 feed portion of the 100-hour test. Deviations from the 100-hour test may include higher feed rates and gas throughputs and the use of a two RP-650T Retech plasma torches to increase throughput and reduce processing time.

The bench-scale furnace has approximately twice the torch power per unit chamber volume compared to either the PCF-6 or the PCF-8. This is offset from a bulk gas temperature standpoint in the bench-scale furnace by the cold wall.

The gas flow through the furnace is a compromise, as higher flows result in lower bulk gas temperatures. The bench-scale furnace gas flow value was selected to approximate the gas flow rate expected in the primary chamber of a PCF, as shown by the volume changes per unit time.

Slag temperature was controlled by the operator, primarily by varying the feed rate and the arc power. Arc power varies with arc length (voltage) and power setting (DC current) as well as other variables. The operator visually verified the fluidity of the slag by observation through the view port to ensure that the slag was thoroughly molten.

The bench-scale furnace is over-powered with respect to torch power per unit slag mass and with respect to torch power per unit slag bed surface area. This allows for a short processing time and provides a conservative test. The validity of this portion of the test was verified by examining the slag product. In the bench-scale furnace, only a small amount of unprocessed soil remained on the bottom of the rotary hearth, near the cold bottom plate. The balance of the slag from the bench-scale furnace was very comparable to the slag produced in a PCF: a homogeneous and monolithic glassy substance.

Expected Correlation of Energy Flows

The bench-scale furnace is also comparable to the PCF on the basis of energy flows. The most important comparison of energy flow is the torch power that flows through the slag and into the rotary hearth or centrifuge circuit, as this allows the slag to reach a fluid state in the PCF. Torch energy flows through the plasma system is a function of the system's construction.

The torch electrode in the RP-75T plasma torch must withstand higher current densities than the electrode in the RP-250T used in the PCF-6 and in the RP-650T used in the PCF-8. This is the reason for higher relative energy flow through the electrode water circuit and the longer life of the larger electrodes. Similarly, the torch ram is smaller in the bench-scale furnace and the bulk gas temperature is lower, which accounts for lower energy flow through the torch ram.

Energy flow through the slag and into the rotary hearth in the bench-scale furnace was within $\pm$5 percent of the energy flow through the centrifuge value for the PCF-8. The 29 percent of torch power value for the centrifuge circuit represents measurements taken on the PCF-8 in Muttenz, Switzerland. The centrifuge on this PCF-8 has air-cooled side walls and thicker insulation than the PCF-6. The PCF-6 centrifuge has water-cooled side walls and thinner insulation, which allows a higher percentage of torch power to flow out through the centrifuge circuit.

Gases leaving the primary chamber account for the largest percentage of torch power in the PCF-8 and the smallest in the bench-scale furnace. This is explained by the largest number of volume changes of gas carrying away energy in the PCF-8 and the larger slag surface exposed to process gas.

Total power equal to 6.36 kW per kg of slag was delivered to the slag in the plutonium volatility test furnace system after all of the material had been fed into the hearth at the end of the test run. This is 25 times the energy required per unit mass to achieve molten and fluid slag in the PCF. The PCF takes approximately 4 hours to achieve operating temperature, and the bench-scale furnace required only approximately 10 minutes. The warm-up time in the PCF is used to achieve a large thermal mass of molten slag within the centrifuge. In the first 10 minutes of the plutonium volatility test, the hearth soil melted and allowed feeding of test soil. Because of the higher relative power available in the bench-scale furnace, the measurement of the volatility of plutonium may be conservative. Because of the operator's ability to adjust the process to achieve slag fluidity and mixing, the results of this test will be representative of PCF operations.

Correlation Summary

The most important correlation between the plutonium volatilization test and the full-scale plasma melter is the fate of plutonium. By correlating the results of cerium sampling and analysis in Tests 1 and 2 and plutonium in Tests 3 and 4 of the plutonium volatilization test

with cerium analysis from the 100-hour operations test, a hypothesis for the fate of plutonium could be developed for a full-scale system. Also, plutonium concentrations reporting to the full-scale off-gas system can be predicted. Correlation calculations are presented in Section 4.2.3..

3.0 TEST PROCEDURE

3.1 Tests 1 through 4

Detailed test procedures are provided in Appendix B. In summary, the loaded hearth was placed in the melt chamber. Feed soil spiked with cerium (for tests 1 and 2) or plutonium (for tests 3 and 4) was loaded into the vibratory feeder and fed to the hearth after all hearth soil was completely melted. In approximately 30 minutes, all soil was fed to the hearth and completely melted. A closed circuit television view of soil melting during test 3 is shown in Figure 3-1.

3.2 Test Runs

Tests 1 and 2 were conducted at the Retech facility in Ukiah, California,, on August 10 and 12, 1993. Tests 3 and 4 were conducted at the Transient Reactor Test Facility (TREAT) at Argonne National Laboratory-West on November 16 and 18, 1993.

3.3 Plutonium Volatilization Sampling and Analysis

Sampling and analytical procedures developed and implemented to achieve the objectives of the plutonium volatilization test are detailed in the Plutonium Volatilization Test Sampling and Analysis Plan (Appendix C). Sample containers ready for test 1 at Retech and test 3 at ANL-W are shown in Figures 3-2 and 3-3. The glovebox that provided access for sampling the cold finger is shown in Figure 3-4.

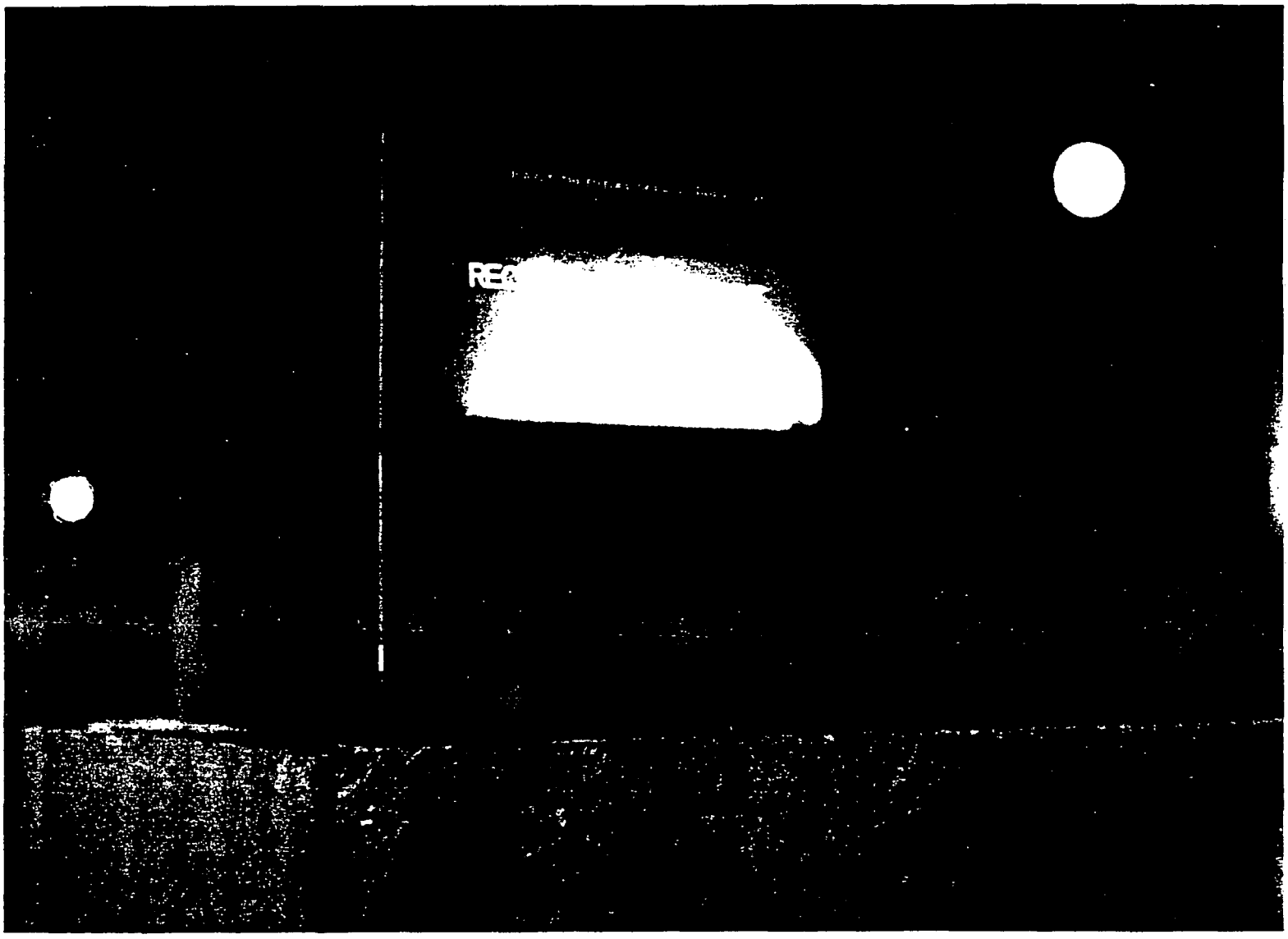

Figure 3-1 CCTV View of Melt, Test 4

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Figure 3-2 Readied Sample Containers, Test 1

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Figure 3-3 Readied Sample Containers, Test 3

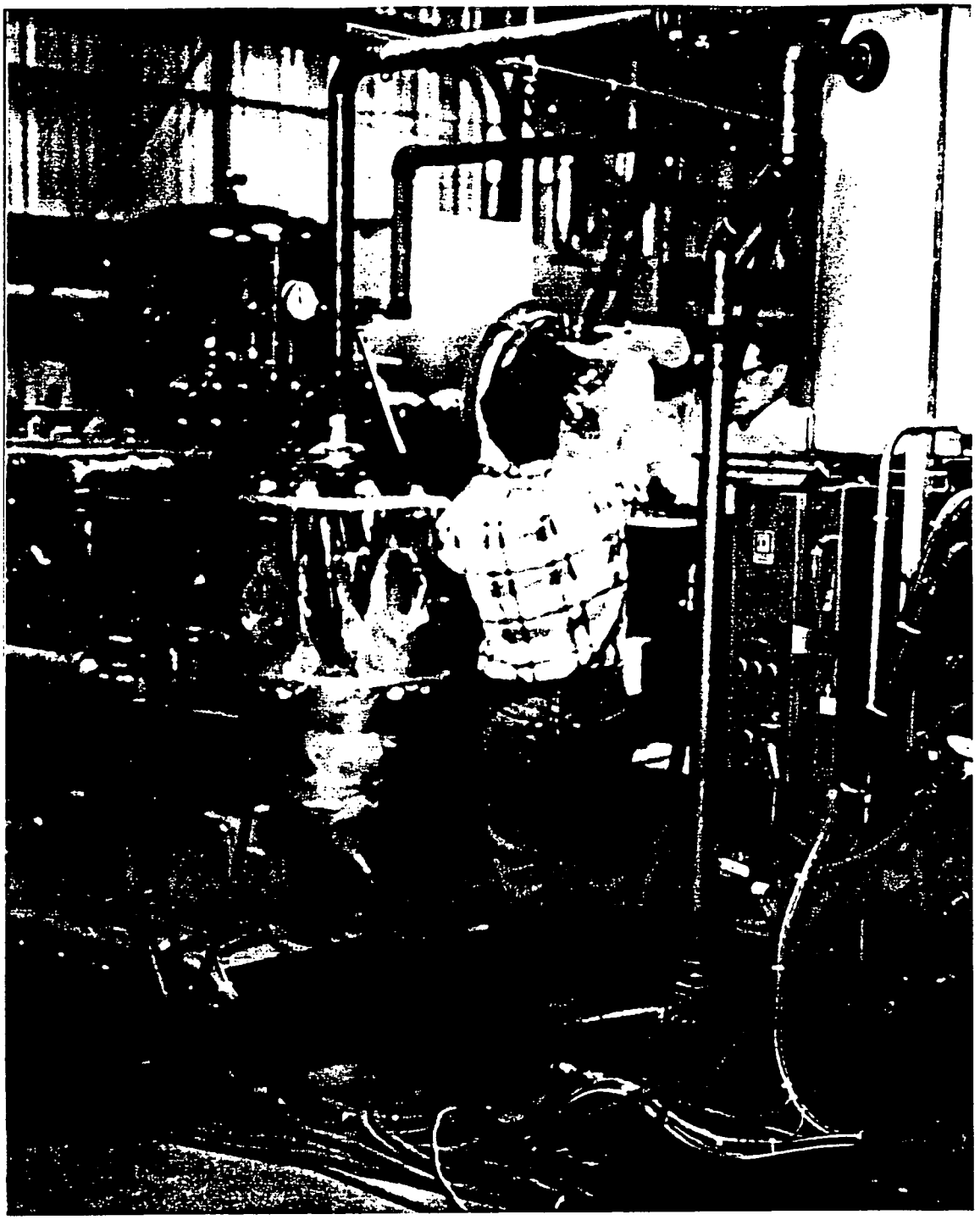

Figure 3-4 Glove Box for Cold Finger Sampling, Tests 3 & 4

4.0 TEST RESULTS

Plutonium volatilization test results described in this section pertain to cerium and plutonium analytical data required to complete analyte mass balances for each of the four tests. Test results are provided in Appendix G.

4.1 Description of Data Generated

Validated plutonium and cerium analytical data as reported by Lockheed Analytical Laboratory (LAL) are presented in Appendix G. Reported data meet all the sampling and analytical completeness goals set forth in the Plutonium Volatilization Test Sampling and Analysis Plan. Analytical data were reported as either a concentration (milligrams [mg] per kilogram, picocuries [pCi] per gram), or total analyte (mg/filter, pCi/sample). To calculate test mass balances, concentrations were converted to total analyte based on test weights as recorded in both sample and operational logbooks.

Quality Assurance personnel from LAL validate all data leaving the laboratory to ensure procedural compliance with the LAL Quality Assurance Plan. The Pit 9 program office then verifies submitted data to ensure sample custody, tracking and analytical requirements have been met. Upon program office verification of slag data generated during Tests 1 and 2 submitted from the laboratory September 1, 1993, it was discovered that the results were consistently an order of magnitude greater than expected. The laboratory then re-verified the slag data and found that the cerium standard preparation as recorded in the standard preparation logbook were prepared at concentrations ten times less than those entered by the analyst as reference standards used in the calculations. Therefore, the data for these analyses were reprocessed using the correct standard concentrations and revised result forms were submitted on October 7, 1993. This factor of ten less error in the standard explains the difference in the results submitted on September 1, 1993 and those submitted on October 7, 1993. A chronology of cerium analysis events prepared by the LAL is provided as Appendix I. Appendix J includes a certification from the Lockheed Data Management Office stating that all data was validated and verified per the Pu Volatilization Sampling and Analysis Plan requirements and all stated data quality objectives were met.

4.2 Data Calculations and Interpretations

Section 4.2.1 below provides a summary of the analytical data; section 4.2.2 provides a discussion of the mass balance analysis.

4.2.1 Analytical Data Summary

Appendix G data are reduced and summarized in Tables 4-1 through 4-14. Laboratory quality control samples analyzed on a per analysis batch basis were used to quantify analytical uncertainty, which was determined to be ±15 percent.

Table 4-1 below presents a summary of background Ce concentrations in the INEL soils used for all Pu Volatilization tests. The mean concentration was calculated and multiplied by the dry weight of Test 1 and 2 soil to determine total background Ce.

Table 4-1: Background INEL Ce Soil Concentrations

Sample ID	Ce Concentration (mg/kg)
P4026A3	78 ± 12
P4027A3	78 ± 12
P4028A3	78 ± 12
P4029A3	69 ± 10
P4030A3	76 ± 11

The mean background Ce concentration was 75.8 mg ± 11.4 mg. The Test 1 dry soil weight was 4522 g and the Test 2 dry soil weight was 4129 g; therefore, the total Test 1 background Ce was 0.34 g ± 0.05 g, and the total Test 2 background Ce was 0.32 g ± 0.04 g. Cerium was added to the soil in the form of an oxide and mixed with test soils as per the Test and Sampling and Analysis plans. Three batches of soil were mixed for each test to allow for re-testing if necessary. The amount of Ce mixed with Test 1 soils was 65.2 g ± 9.8 g and 54.3 g ± 8.1 g for Test 2 soils. The total input for the tests was therefore the sum of background and added Ce; for Test 1 the total Ce input was 65.5 g ± 9.8 g and for Test 2 the total Ce input was 54.6 g ± 8.1 g.

Table 4-2 below summarizes Test 1 slag analysis for Ce. Data is presented as a concentration (percent Ce). Total Ce in slag was determined by multiplying the concentration by the total Test 1 slag weight as recorded in the sample logbook (note: 1% = 10,000 mg/kg).

The Test 1 slag weight was 3625 g. The mean Ce concentration in the slag was 1.4% ± 2100 mg/kg, or 14,000 mg/kg ± 2100 mg/kg. The total Ce in the Test 1 slag was therefore 50.7 g ± 7.6 g.

Table 4-2: Test 1 Slag Data Summary

Sample ID	Ce Concentration (%)
P4040A3	1.5 ± 2250 mg/kg
P4041A3	1.5 ± 2250 mg/kg
P4042A3	0.9 ± 1350 mg/kg
P4043A3	1.5 ± 2250 mg/kg
P4044A3	1.6 ± 2400 mg/kg

Table 4-3 below presents a summary of data collected from Test 1 melt chamber material. This includes wipes of all melt chamber components as well as spill and feeder residual and hearth refractory. Uncertainty values less than 10 mg are insignificant to the system mass balance and not included in this table.

Table 4-3: Test 1 Melt Chamber Data Summary

Sample ID	Sample Description	Total Ce (mg)
P4055A3	Rear Wall Wipe	0.5
P4056A3	Door Wipe	0.5
P4201A3	Wall Debris	77.0 ± 11
P4054A3	Torch Ram Wipe	0.4
P4053A3	Wall Wipe	2.9
P4202A3	Torch Ram Insulator	3.8
P4203A3	Hearth Refractory	5.6
P4046A3	Feed Spill	1400 ± 210
P4215A3	Feed Residual	1620 ± 243

Feed spill and residual data analysis are included in the system mass balance. Total Ce from the melt chamber is the sum of all components above not including feed and spill residual. Total Test 1 melt chamber Ce was 90.7 mg or 0.091 g ± 0.01 g.

Table 4-4 below summarizes data collected from each component of the off gas system. Units are in micrograms; uncertainty values less than 10 mg are insignificant to the system mass balance and not included in this table.

Table 4-4: Test 1 Off Gas System Data Summary

Sample ID	Sample Description	Total Ce (µg)
P4057A3	Cold Finger Wipe	3100
P4058A3	Cold Finger Wipe	330
P4204A3	Off Gas Wipe	16
P4048A3	HEPA	29/sample
P4049A3	HEPA	39/sample
P4050A3	HEPA	27/sample
P4052A3	HEPA	32/sample

Each HEPA filter sample was approximately 2 in^3. The HEPA filter data indicate an even distribution of Ce throughout the entire HEPA (729 in^3). Based on a mean of 32 µg Ce/2 in^3, the total amount of Test 1 HEPA Ce was 11.7 mg. Contributions from other off gas components amount to 3.4 mg Ce. Total off gas Ce is therefore 15.1 mg or 0.015 g.

Table 4-5 below summarizes Test 2 slag analysis for Ce. Data is presented as a concentration (percent Ce). Total Ce in slag was determined by multiplying the concentration by the total Test 2 slag weight as recorded in the sample logbook (note: 1% = 10,000 mg/kg).

Table 4-5: Test 2 Slag Data Summary

Sample ID	Ce Concentration (%)
P4060A3	1.3 ± 1950 mg/kg
P4061A3	1.2 ± 1800 mg/kg
P4062A3	1.3 ± 1950 mg/kg
P4063A3	1.3 ± 1950 mg/kg
P4064A3	1.4 ± 2100 mg/kg

The Test 2 slag weight was 3815 g. The mean Ce concentration in the slag was 1.3% ± 1950 mg/kg, or 13,000 mg/kg ± 1950 mg/kg. The total Ce in the Test 2 slag was therefore 49.6 g ± 7.4 g.

Table 4-6 below presents a summary of data collected from Test 2 melt chamber material. This includes wipes of all melt chamber components as well as spill and feeder residual and hearth refractory.

Figure 4-6: Test 2 Melt Chamber Data Summary

Sample ID	Sample Description	Total Ce (mg)
P4074A3	Rear Wall Wipe	2.2
P4075A3	Door Wipe	1.8
P4210A3	Wall Debris	121 ± 18
P4073A3	Torch Ram Wipe	6.2
P4076A3	Wall Wipe	2.2
P4212A3	Hearth Refractory	5.6
P4066A3	Spill and Feed Residual	6300 ± 945

Error bands are not stated for uncertainty values less than 10 mg. Spill and feed and residual data analysis are included in the section 4.2.2 system mass balance. Total Ce from the melt chamber is the sum of all components above not including spill and feed residual. Total Test 2 melt chamber Ce was 139 mg or 0.139 g ± 0.02 g.

Table 4-7 below summarizes data collected from each component of the Test 2 off gas system. Units are in micrograms. Uncertainty values less than 10 mg are insignificant to the system mass balance and not included in this table.

Each HEPA filter sample was approximately 2 in^3. The HEPA filter data indicate a fairly even distribution of Ce throughout the entire HEPA (729 in^3). Based on a mean of 49 µg Ce/2 in^3, the total amount of Test 2 HEPA Ce was 17.9 mg. Contributions from other off gas components amount to 3.6 mg Ce. Total off gas Ce is therefore 21.5 mg or 0.021 g.

Table 4-7: Test 2 Off Gas Data Summary

Sample ID	Sample Description	Total Ce (µg)
P4077A3	Cold Finger Wipe	2200
P4078A3	Cold Finger Wipe	630
P4214A3	Cold Finger Debris	720
P4213A3	Off Gas Wipe	35
P4068A3	HEPA	50/sample
P4069A3	HEPA	48/sample
P4071A3	HEPA	47/sample
P4072A3	HEPA	53/sample

Plutonium for Tests 3 and 4 was leached from Rocky Flats Plant Soil and mixed as a slurry with "clean" INEL soils. Sampling and analysis uncertainty is primarily attributed to radiological analysis error resulting from residual background instrumentation levels, count times, and sample volume. The activity uncertainty is unique to each sample and can be found under the heading of "error" on the radionuclide analysis reports in Appendix G. Five samples were collected for analysis to determine Test 3 and 4 input Pu. Test 3 and 4 feed soil data is summarized in Table 4-8 below.

Table 4-8: Test 3 and 4 Feed Soil Data Summary for Plutonium:

Sample ID	Pu Activity (pCi/g)
P4033A3	1580 ± 116
P4034A3	2452 ± 230
P4035A3	2165 ± 190
P4036A3	2303 ± 200
P4037A3	2352 ± 199

The mean activity of Test 3 and 4 soils was 2170 pCi/g ± 187 pCi/g. The weights of the Test 3 and 4 feed soils were 2595 g and 3621 g, respectively. Total Test 3 input Pu activity was 5.63×10^6 pCi ± 0.48×10^6 pCi. Total Test 4 input Pu activity was 7.86×10^6 ± 0.68×10^6 pCi.

Table 4-9 below summarizes Test 3 slag analysis for Pu. Total Pu in slag was determined by multiplying the mean concentration by the total Test 3 slag weight as recorded in the sample logbook.

Table 4-9: Test 3 Slag Data Summary

Sample ID	Pu Activity (pCi/g)
P4080A3	1208 ± 163
P4081A3	1835 ± 236
P4082A3	1381 ± 155
P4315A3	1728 ± 219
P4316A3	1693 ± 153

The Test 3 slag weight was 4074 g. The mean Pu activity in the slag was 1569 pCi/g ± 185 pCi/g. The total Pu activity in the Test 4 slag was therefore 6.39×10^6 pCi/ g ± 0.75×10^6 pCi/ g.

Table 4-10 below presents a summary of Pu data collected from Test 3 melt chamber material. This includes wipes of all melt chamber components as well as spill and feeder residual and hearth refractory.

Figure 4-10: Test 3 Melt Chamber Data Summary

Sample ID	Sample Description	Total Pu (pCi)
P4093A3	Rear Wall Wipe	99±15
P4096A3	Door Wipe	102±15
P4092A3	Wall Debris	0.115×10^5± 1728
P4094A3	Torch Ram Wipe	155±23
P4095A3	Wall Wipe	616±92
P4314A3	Torch Ram Insulator	1073±161
P4317A3	Hearth Refractory	<MDA
P4091A3	Spill and Feed Residual	1.77×10^5± 0.26×10^5

Spill and feed and residual data analysis are included in the section 4.2.2 system mass balance. Total Pu from the melt chamber is the sum of all components above not including spill and feed residual. Total Test 3 melt chamber Pu was 0.136×10^5 pCi ± 2035 pCi.

Table 4-11 below summarizes data collected from each component of the Test 3 off gas system. Units are in picocuries.

Table 4-11: Test 3 Off Gas Data Summary

Sample ID	Sample Description	Total Pu Activity (pCi)
P4302A3	Cold Finger Wipe	149 ± 22
P4301A3	Cold Finger Wipe	26 ± 4
P4300A3	Cold Finger Debris	774 ± 116
P4303A3	Off Gas Wipe	<MDA
P4086A3	HEPA	2 pCi/sample
P4087A3	HEPA	3 pCi/sample
P4088A3	HEPA	2 pCi/sample
P4089A3	HEPA	2 pCi/sample
P4090A3	HEPA	4 pCi/sample

Each HEPA filter sample was approximately 2 in^3. Uncertainty values for the HEPA samples were insignificantly small relative to mass balance values and are not included in the table. The HEPA filter data indicate a fairly even distribution of Ce throughout the entire HEPA (729 in^3). Based on a mean of 2.6 pCi Pu/2 in^3, the total Pu activity of the Test 3 HEPA was 948 pCi ± 142 pCi. Contributions from other off gas components amount to 949 pCi ± 142 pCi. Total off gas Pu activity is therefore 1897 pCi ± 284 pCi.

Table 4-12 below summarizes Test 4 slag analysis for Pu. Total Pu in slag was determined by multiplying the mean concentration by the total Test 4 slag weight as recorded in the sample logbook.

The Test 4 slag weight was 5183 g. The mean Pu activity in the slag was 1661 pCi/g ± 196 pCi/g. The total Pu activity in the Test 4 slag was therefore 8.61×10^6 pCi/ g ± 1.02×10^6 pCi/ g.

Table 4-12: Test 4 Slag Data Summary

Sample ID	Pu Activity (pCi/g)
P4318A3	1542 ± 180
P4319A3	1673 ± 218
P4098A3	1715 ± 200
P4099A3	1677 ± 189
P4100A3	1699 ± 192

Table 4-13 below presents a summary of Pu data collected from Test 3 melt chamber material. This includes wipes of all melt chamber components as well as spill and feeder residual and hearth refractory.

Figure 4-13: Test 4 Melt Chamber Data Summary

Sample ID	Sample Description	Total Pu (pCi)
P4111A3	Rear Wall Wipe	111 ± 17
P4114A3	Door Wipe	118 ± 17
P4110A3	Wall Debris	0.178×10^5 ± 2670
P4112A3	Torch Ram Wipe	120 ± 18
P4113A3	Wall Wipe	207 ± 31
P4314A3	Torch Ram Insulator	1073 ± 161
P4320A3	Hearth Refractory	0.30×10^5 ± 4500
P4109A3	Spill and Feed Residual	2.81×10^5 ± 4215

Spill and feed and residual data analysis are included in the section 4.2.2 system mass balance. Total Pu from the Test 4 melt chamber is the sum of all components above not including spill and feed residual. Total Test 4 melt chamber Pu was 0.494×10^5 pCi ± 7414 pCi.

Table 4-14 below summarizes data collected from each component of the Test 4 off gas system. Units are in picocuries.

Table 4-14: Test 4 Off Gas Data Summary

Sample ID	Sample Description	Total Pu Activity (pCi)
P4311A3	Cold Finger Wipe (housing)	71 ± 11
P4312A3	Cold Finger Wipe	343 ± 51
P4310A3	Cold Finger Debris	92 ± 14
P4313A3	Off Gas Wipe	<MDA
P4104A3	HEPA	5 pCi/sample
P4105A3	HEPA	4 pCi/sample
P4106A3	HEPA	6 pCi/sample
P4107A3	HEPA	5 pCi/sample
P4108A3	HEPA	5 pCi/sample

Each HEPA filter sample was approximately 2 in^3. Uncertainty values for the HEPA samples were insignificantly small relative to mass balance values and are not included in the table. Conservatively assuming an equal distribution of Pu throughout the entire HEPA (729 in^3) and based on a mean of 5 pCi Pu/2 in^3, the total Pu activity of the Test 4 HEPA was 1822 pCi ± 273. Contributions from other off gas components amounted to 506 pCi ± 76 pCi. Total off gas Pu activity is therefore 2328 pCi ± 349 pCi.

4.2.2 Mass Balance Analysis

For purposes of calculating the cerium mass balance, data were grouped into the following data summaries:

- INEL background cerium soil concentrations
- Slag cerium concentrations
- Melt chamber total cerium
- Off-gas system total cerium

The cerium mass balance was calculated as follows:

$$Ce_{in} = Ce_{out}$$

$$Ce_{in} = Ce_{background} + Ce_{spiked} - Ce_{feed\ residual}$$

$$Ce_{out} = Ce_{slag} + Ce_{melt\ chamber} + Ce_{off\ gas}$$

As shown in Table 4-15, the Test 1 oxidizing environment resulted in 99.81% ± 15.0% of the cerium bound with the slag, with 0.18% ± 0.02% dispersed within the melt chamber and 0.03% ± 0.02% detected in the off-gas system, primarily the HEPA filter. The Test 2 reducing environment resulted in 99.67% ± 14.9% of the cerium bound with the slag, 0.28% ± 0.04% percent in the melt chamber, and 0.28% ± .02% in the off-gas system. The slightly lower percentage of cerium in the Test 2 slag may be the result of slightly greater cerium volatilization, or it may have resulted from a more turbulent melt chamber environment caused by the volatilization of the spiked PVC.

Tests 3 and 4 plutonium mass balance was calculated exactly as the cerium mass balance. There are no background plutonium levels in the INEL soil.

$$Pu_{in} = Pu_{out}$$

$$Pu_{in} = Pu_{spiked} - Pu_{feed\ residual}$$

$$Pu_{out} = Pu_{slag} + Pu_{melt\ chamber} + Pu_{off\ gas}$$

As shown in Table 4-15, the Test 3 oxidizing environment resulted in 99.84% ± 11.7% of the plutonium bound with the slag, 0.16% ± 0.16% dispersed within the melt chamber, and 0.16% ± 0.16% detected in the off-gas system, primarily the HEPA filter. The Test 4 reducing environment resulted in 99.4% ± 11.8% of the plutonium in the slag, 0.58% ± 0.12% in the melt chamber, and 0.12% ± 0.12% in the off-gas system.

The cerium and plutonium mass balances are summarized in Table 4-16. For Tests 1 and 2, the cerium mass closures are 81.3% ± 20.4% and 103% ± 24.1%, respectively. For Tests 3 and 4, the plutonium mass balances are 117.4% ± 16.3% and 114% ± 22%, respectively.

Tests 2 and 4 reducing environment resulted in slightly greater cerium/plutonium dispersed within the melt chamber. As discussed briefly above, these slightly higher analyte levels can be attributed to increased volatilization resulting from high-temperature chemistry in a reducing environment, increased particle entrainment caused by PVC volatilization turbulence, or a combination of the two. Regardless, it can be concluded that greater than 99 percent of all available cerium and plutonium became bound with the slag, which correlates well with results from the 100-hour operations test, as discussed in section 4.2.3.

Table 4-15: Ce/Pu Test Output Streams by Percent

Output Stream	Test 1 Cerium	Test 2 Cerium	Test 3 Plutonium	Test 4 Plutonium
Slag	99.81% ± 15.0%	99.67% ± 14.9%	99.84% ± 11.7%	99.40% ± 11.8%
Melt Chamber	0.18% ± 0.02%	0.28% ± 0.04%	0.16% ± 0.16%	0.58% ± 0.12%
Off Gas	0.03% ± 0.02%	0.28% ± 0.02%	0.16% ± 0.16%	0.12% ± 0.12%

Table 4-16: Pu Volatilization Mass Balance Summary Table

Source		Test 1 Cerium (grams)	Test 2 Cerium (grams)	Test 3 Plutonium (pCi x 10^6)	Test 4 Plutonium (pCi x 10^6)
Input	Soil Background	0.34 ± 0.05	0.32 ± 0.04	0	0
	Surrogate Addition	65.2 ± 9.8	54.3 ± 8.1	5.63 ± 0.48	7.86 ± 0.67
	Feeder/Spill Residual (-)	3.0 ± 0.4	6.3 ± 0.9	0.18 ±	0.28 ±
	Input Total	62.5 ± 10.2	48.3 ± 9.0	5.45 ± 0.48	7.58 ± 0.67
Output	Slag	50.7 ± 7.6	49.6 ± 7.4	6.39 ± 0.75	8.61 ± 1.02
	Melt Chamber	0.09 ± 0.01	0.14 ± 0.02	0.01 ± 0.01	0.05 ± 0.01
	Off Gas	0.02 ± 0.01	0.02 ± 0.01	< 0.01 ± 0.01	< 0.01 ± 0.01
	Output Total	50.1 ± 7.6	49.8 ± 7.4	6.40 ± 0.75	8.66 ± 1.02
	Mass Closure	**81.3% ± 20.4%**	**103% ± 24.1%**	**117.4% ± 16.3%**	**114% ± 22%**

4.2.3 Correlation with the 100-Hour Operations Test

The intent of correlating data from the plutonium volatilization and 100-hour operations test is to predict approximate plutonium emissions resulting from full-scale Pit 9 remediation. The credibility of this correlation is strengthened by the consistency of data derived independently from both tests. The correlation is as follows:

$$\frac{\%Ce\ volatilized_{Pu\ volatilization}}{\%Ce\ volatilized_{100\text{-}hr}} = \frac{\%Pu\ volatilized_{Pu\ volatilization}}{\%Pu\ volatilized_{100\text{-}hr}\ (unknown)}$$

"Volatilized analyte" is that detected in the off-gas system. Off-gas system data from the plutonium volatilization test are presented in Section 4.2.1. Off-gas system data from the 100-hour test are derived for cerium concentrations analyzed from samples of the three scrubber water tanks, scrubber water filtrate, cyclone particulate, baghouse shakedown, and in-line water filters. The total cerium detected in the 100-hour off-gas system was 1.8 g; input cerium totaled 42,162 g. Therefore, 0.004 percent of available Ce was detected in the 100-hour off-gas system. (The uncertainty in the cerium in the off gas system from the 100-hr test was less than 10 mg, so no uncertainty value is available for this result.) Completing the correlation:

$$\frac{0.04\% \pm 0.02\%}{0.006\%} = \frac{0.16\% \pm 0.16\%}{x}, \qquad x = 0.016\% \pm 0.016\%$$

Hypothesizing that based on these results, 0.016 percent of all available Pit 9 plutonium will report to the full-scale off-gas system and 30 kg of plutonium are buried in Pit 9, then (0.016 percent)(30 kg) or 4.8 g of plutonium will enter the off-gas system over the duration of the project. Assuming double HEPA banks and a worst-case condition of failure of upstream off-gas treatment and all available plutonium reporting directly to the HEPAs at once, then 4.3×10^{-7} g of plutonium will be emitted to the atmosphere.

In-situ vitrification (ISV) tests on Pu spiked soils resulted in 0.022% Pu released into the off-gas (KM Oma and CL Timmerman, "Off-Gas Treatment and Characterization for a Radioactive In-Situ Vitrification Test", PNL-SA-12000, Pacific Northwest Laboratories, August 1984). The predicted results of plutonium volatilized from the 100-hr melter test (0.016% ± 0.017%) are comparable to the ISV results within the experimental uncertainties. More significantly, the amount of Pu volatilized is within the removal capacity of an off-gas cleanup system designed to meed DOE Order 6430.1A requirements, and no additional plans for melter off-gas Pu removal are required.

5.0 TEST CONCLUSIONS

The following conclusions are based on results from the plutonium volatilization test:

- Under both oxidizing and reducing environments, plutonium and cerium are volatilized into the off-gas system in concentrations less than 0.5 percent.
- Less than one microgram of plutonium is expected to be released from the off-gas system to the environment during the entire Pit 9 remediation.

Appendix I

Chronology of Cerium Analysis

Appendix J

Data Validation and Verification Certification

LIST OF FIGURES

LIST OF TABLES